



Designation: F3169 – 16

# Standard Test Method for Leak Detection in Blister Packaging by Vacuum Deflection Method by Laser Measurement<sup>1</sup>

This standard is issued under the fixed designation F3169; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 *Test Packages*—This test method can be applied to non-porous blister packs sealed with flexible films such as those used in pharmaceutical packaging. Such blister packs typically consist of thermoformed polymer or cold formed aluminum trays that contain a number of individual blister pockets into which tablets or capsules are placed. The trays are then sealed with a polymer, paper-backed or foil-based flexible laminate lidding material.

1.2 *Leaks Detected*—This test method detects leaks in blister packs by measuring the deflection of the blister pack surface in response to an applied vacuum. This deflection of the blister pack surface results from the difference in pressure between the gas inside the blister pack and the applied vacuum. Air loss from within a blister pocket as a result of a leak will alter this pressure differential causing a measureable variation in blister pocket deflection. This test method requires that the blister packs are held in appropriate tooling inside a suitable test chamber.

1.3 *Test Results*—Test results are reported qualitatively (pass/fail). Appropriate acceptance criteria for *deflection*, *height*, and *collapse* values are established by comparing non-leaking packs with those containing defects of a known size. Suitably sized defects in the laminate, tray material, and seal can be detected using this test method. The sensitivity of this test method depends upon a range of factors including blister pocket headspace, blister pocket size, lidding material type, lidding material thickness, lidding material tension, printing, surface texture, test conditions, and the values selected for the pass/fail acceptance criteria. The ability of the test to detect 15  $\mu\text{m}$ , 50  $\mu\text{m}$ , and catastrophic sized holes in four blister pack designs was demonstrated in a study.

1.4 The values stated in SI units are to be regarded as standard and no other units of measurement are included in this test method.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards*:<sup>2</sup>

**D996 Terminology of Packaging and Distribution Environments**

**F17 Terminology Relating to Flexible Barrier Packaging**

## 3. Terminology

3.1 For definitions used in this test method, see Terminologies **D996** and **F17**.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *blister pack, n*—forming material, encapsulated product, and sealed lidding material.

3.2.2 *blister pocket, n*—sealed cavity in the forming material that contains product.

3.2.3 *collapse, n*—difference in height of the blister pocket profile in one plane before and after the reduced vacuum has been applied.

3.2.4 *deflection, n*—difference in height of the blister pocket profile in one plane before and after the initial vacuum has been applied.

3.2.5 *height, n*—difference in height of the blister pocket profile in one plane after initial vacuum when compared against blister pack surface height.

3.2.6 *profile, n*—surface of the lidding material measured at regular intervals across the blister pocket.

## 4. Summary of Test Method

4.1 The test blister packs are located in the test chamber in appropriate tooling to prevent them from moving under vacuum. The chamber is then sealed and the profile of every

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

blister pocket is measured using an appropriate measurement device such as a laser and actuator (see Fig. 1).

4.2 A vacuum is then applied to the test chamber and each blister pocket measured again. Sealed blister pockets can be identified by the change in measurement between the first and second profiles. This is known as *deflection* and is caused by the pressure difference between the air inside the blister pocket and the air in the chamber caused by the application of vacuum (see Fig. 2).

4.3 The *deflection* of a blister pocket with a gross hole will be minimal as the pressure difference between the air inside and outside of the blister pocket will equalize through the hole.

4.4 The sensitivity of the test can be increased by holding the test samples under vacuum for a period of time and then reducing the vacuum level. After reducing the vacuum, the surface profile of the blister pockets can be measured again and compared with the profiles recorded after the initial vacuum was applied. Reducing the vacuum level causes blister pockets with small holes to have a collapse measurement greater than blister pockets with no defects. This is due to air leaking from any small defects (see Fig. 3).

4.5 The sensitivity of the test is a function of the test parameters chosen, namely vacuum levels, hold time, and the *pass/fail* criteria selected. Blister pack design including head-space and blister pocket size, lidding material, lidding material thickness, lidding material tension, printing, and surface texture can affect test sensitivity. The vacuum levels used are typically in the range of 30 - 60 KPa and are chosen based on setup tests conducted using good blister packs and those containing defects of known size. The required vacuum level depends upon the blister pack design and materials. For example, detecting sub-15  $\mu\text{m}$  defects in small blister pockets sealed with a stiff lidding material typically requires vacuum levels towards the higher end of the standard range. The vacuum level is then reduced and the packs are rescanned after a holding period (typically 10 - 45 s) at this reduced vacuum. The test parameters, including the *pass/fail* criteria, are chosen

by comparing the *deflection* and *collapse* values of good blister packs and those containing defects of the required size. Such defects can be produced by using a wire, by laser drilling, or by applying self-adhesive predrilled holes.

4.6 Where blister pockets appear pre-swollen the flat sealed surface of the blister pack is used to project a datum line across the blister pocket from which a change in height under vacuum can be observed. If this height value is small or negative it can be understood that the blister pocket has a gross hole, as the blister pocket has not deflected due to vacuum or positive pressure before testing (see Fig. 4).

4.7 Any blister pocket that falls outside the acceptance criteria for *deflection*, *collapse*, or *height* is recorded as a fail. The ability of the test to detect 15 and 50  $\mu\text{m}$  sized laser drilled holes was verified using four blister pack designs. This method was also used to find large catastrophic defects demonstrated with a 5 mm slit in the lidding material.

4.8 Background noise may occur due to movement of the blister pack under vacuum. Such noise can be minimized by selection of an appropriate measurement device, proper retention of the blister pack, and careful selection of pressures used for testing.

## 5. Significance and Use

5.1 Leaks in blister packs may affect product quality and such defects can arise from imperfections in the packaging material or bond between the sealed surfaces.

5.2 This method of leak testing is a useful tool as it allows non-destructive and non-subjective leak testing of blister packs. It allows the operator to evaluate how different packaging materials and packaging machine conditions affect the integrity of the packaging. It can also provide indication of unwanted changes in the packaging conditions.

5.3 This type of testing is typically used in pharmaceutical packaging production, during stability trials and for package research and development operations because of its non-destructive nature, cleanliness, and speed.

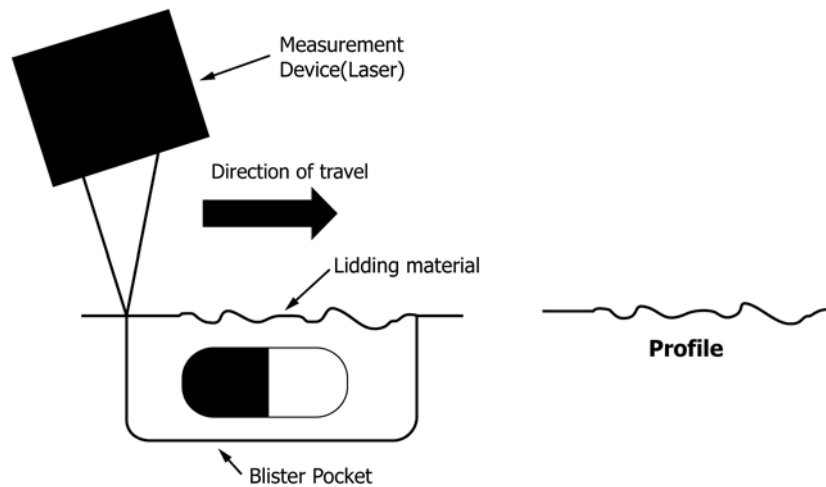


FIG. 1 Profile

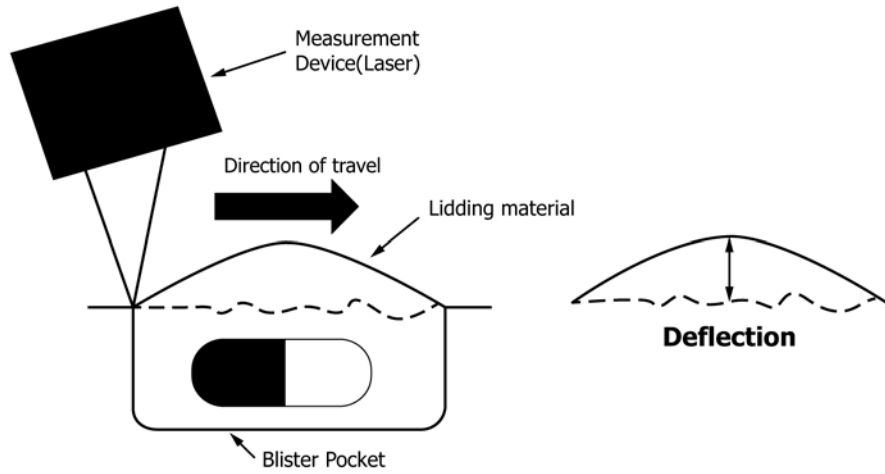


FIG. 2 Deflection

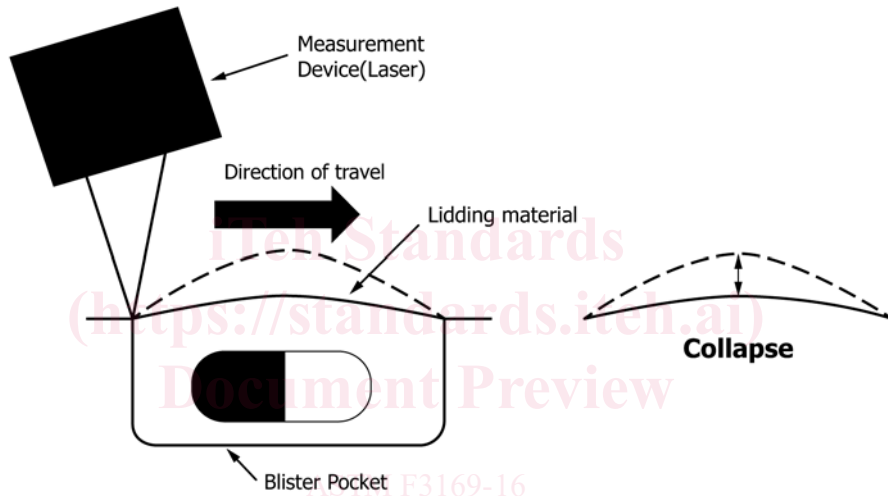


FIG. 3 Collapse

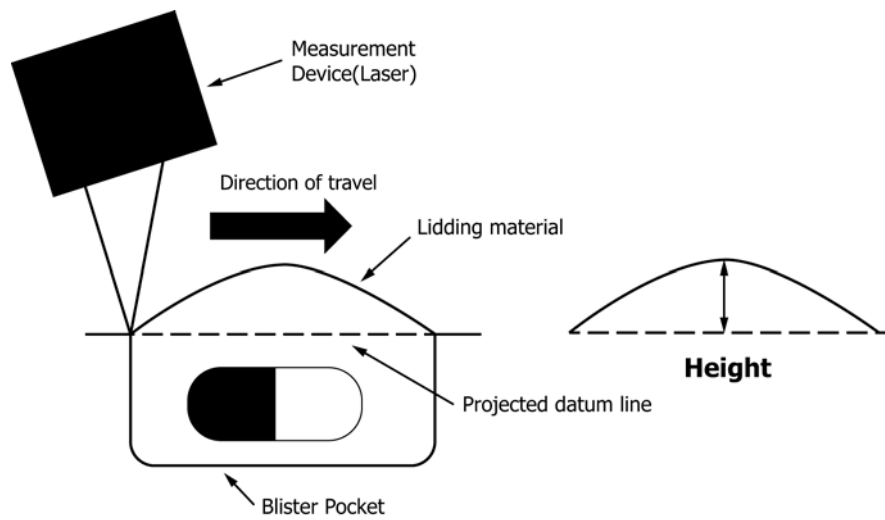


FIG. 4 Height

## 6. Apparatus

6.1 The leak testing instrumentation consists of a vacuum chamber and measurement device (see Fig. 5).

6.2 *Test Chamber*—The test chamber maintains the blister packs under vacuum, typically at levels between 30 - 60 kPa during the test cycle. It locates the lower and upper tooling,